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# Extractable DOC and DON from a dry-land long-term rotation and cropping system in Texas, USA

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ABSTRACT

Soil solution chemistry may be responsible for the solubility and subsequent losses of DOC and DON from terrestrial ecosystems. The objective of this research was to determine whether there was a significant difference in recoverable soil dissolved organic carbon (DOC), organic nitrogen (DON), and dissolved inorganic nitrogen (DIN) extracted with cold and hot water, 10 mM $CaCl_2$, 2 M KCl and 0.5 M $K_2SO_4$ in four long-term agricultural tillage and cropping management systems and whether the type of extractant, tillage or cropping regime was responsible for decoupling the relationship between DOC and DON. The recovery of DOC and DON from soils was highly dependent upon the method of extraction. 10 mM $CaCl_2$ recovered the least DOC, which ranged from 49 to 139 μg $g^{-1}$, while 0.5 M $K_2SO_4$ recovered the most DOC and ranged from 176 to 290 μg $g^{-1}$. Hot water extracted significantly greater DON than any of the other extractants among all cropping and tillage systems. The relationship between DON and DOC was strong and significant in hot and cold water extracts ($R^2 = 0.94$ and 0.55 respectively), but their relationship was decoupled with chemical extractants and a continuous cropping.

## 1. Introduction

Dissolved organic carbon (DOC) and dissolved organic nitrogen (DON) are recognized as important components of soil microbial processes and subsequent nutrient cycling in soils. Dissolved organic carbon (DOC) comprises a continuum of small organic molecules to highly polymeric humic substances and is typically defined as material that passes through a 0.45 μm filter (Thurman, 1985). Dissolved organic nitrogen is a subset of the DOC pool comprising amino acids, proteins and peptides (Leenheer, 2004). DON is typically strongly related to DOC in undisturbed ecosystems (McDowell, 2003). Recently, occurrences of DOC and DON decoupling have been reported from different land uses. Inamdar and Mitchell (2007) reported decoupling of DOC and DON in a wetland and postulated it was due to increased saturation from storm events. Flowpath of water through a watershed favoring organic rather than mineral soil layers may be responsible for decoupling DOC and DON in forested watersheds of Nova Scotia (Aitkenhead-Peterson et al., 2005). Soil nitrogen status can result in increased DON but not DOC resulting in a decoupling of the two organic fractions (McDowell et al., 2004). Addition of lime to a forest soil to mitigate the effects of acid rain resulted in a decoupling of DOC and DON in Swedish forests (Andersson et al., 1999).

Because of the expense of producing, installing and maintaining zero tension lysimeters, many researchers choose to extract or leach soils to obtain DOC and dissolved organic N (DON) extracts as a surrogate for soil solution. The concentration of extracted DOC and DON is however often very different relative to DOC in leachate collected by field lysimeters or DOC obtained using in vitro leaching (Chantigny, 2003). MacDonald et al. (2004) suggested that by leaching air-dried, sieved (4 mm), soil in columns with ultra pure water until electrical conductivity (EC) was in equilibrium, the soils, when leached with a solution comprising a weak salt content, mimicked DOC concentrations found in leachate collected by field lysimeters. Jones and Willett (2006) also pointed out that extracted DOC and DON do not accurately reflect a soils' DOC and DON status and suggested that concentrations should be monitored throughout the year to take into account both seasonal and short-term climatic effects.

Many studies have presented results of extractable DOC albeit using a diverse range of extractants on soils from a range of land uses (e.g. Dou et al., 2007, 2008a,b; Jones and Willett, 2006; Suominen et al., 2003; Wright et al., 2007; Zsolnay and Gorlitz, 1994). Chantigny (2003) concluded after examining a large dataset of dissolved organic matter (DOM) collected from ecosystems by various methods that it was difficult to make generalizations due to changes in land use and management practices because the methodologies used to measure extractable organic matter were not comparable to each other.

Numerous types of extractants, such as distilled water, 4 mM $CaSO_4$, 0.5 M $K_2SO_4$, and 2 M KCl, are often used as extractants making it very difficult to compare and contrast across land uses (Chantigny,

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2003; Jones and Willett, 2006; Willett et al., 2004). Not only are the types of extractants important but shaking time and intensity, water to soil ratio used and temperature can make a difference in the amount of DOC and DON recovered from soil (Jones and Willett, 2006; Willett et al., 2004). While Jones and Willett (2006) recommended the development of a standardized extraction method, which is a necessity to reduce uncertainty when comparing the results of different studies, they also found that different soil types may respond very differently to extractants and possibly confound method standardization.

The majority of studies investigating soil solution or extractable DOC and DON have occurred in forested or rangeland systems (e.g. Jones and Willett, 2006; McDowell et al., 2004; Qualls et al., 2000). Fewer studies have examined DOC or DON in agricultural systems under differing cropping and tillage systems (e.g. Dou et al., 2007, 2008a,b; Wright et al., 2007) or from soil under grasslands (Jones and Willett, 2006). The objective of this study was three-fold, a) to compare extractable DOC, DON, ammonium-N and nitrate-N among four long-term cropping and rotation treatments, b) to compare extraction methods and leached soil solution for the retrieval of DOC and total dissolved nitrogen (TDN) and c) determine if the use of chemical extractants (salts), cropping or tillage had an effect on the strong relationship between DON and DOC typically seen in undisturbed systems (McDowell, 2003).

## 2. Material and methods

### 2.1. Site description

The samples used in this study were collected from a dryland farming system trial initiated in 1982 in south-central Texas situated at the Texas A&M University Research Farm near College Station, TX (30°32′N, 94°26′W). The soil at this site is a Weswood silty clay loam and has been characterized as fine silty, mixed, thermic Fluventic Ustochrept (Franzluebbers et al., 1995). Soil contained 45% silt, 43% clay and 12% sand (Dou et al., 2008a). Two tillage regimes and two cropping systems were sampled in our study. The treatments included: 1) no-till, continuous wheat (NTCW; *Triticum aestivum* L.); 2) conventional till, continuous wheat (CTCW); 3) no-till, sorghum (*Sorghum bicolor* L. Moench), wheat and soybean rotation (NTRW; *Glycine max* L. Merr); and 4) conventional till, sorghum, wheat and soybean rotation (CTRW). Treatment plots were 4×12.2 m and were replicated four times. Plots with continuous wheat (CW) and rotated wheat (RW) treatments received annual broadcasted N fertilizer ($NH_4NO_3$) at 68 kg N $ha^{-1}$. Conventional tillage (CT) operations consisted of disking (100 to 150 mm depth) after harvest, followed by chisel-plowing (200 to 250 mm depth) with a second disking, and ridging prior to winter. Under the no-tillage (NT) treatments, no soil disturbance occurred except for banded fertilizer application to sorghum and planting of crops (González-Chávez et al., 2010).

### 2.2. Soil sampling

Random soil cores (2-cm diameter, 5 cm depth) were collected and composited to form 16 samples (1 composite sample from each plot). Soil cores were collected near the beginning of wheat growth. Wheat was planted in 0.18-m-wide rows at ca. 9.3 g $m^{-2}$ in early November 2007, and soil samples were taken in late November 2007. Soil samples were placed in Ziploc plastic bags and stored in a cooler immediately upon sampling and then sieved moist (2 mm), mixed well to homogenize, and stored at 4 °C until extraction the next day.

### 2.3. Preparation of soil extracts

To allow direct comparison between treatments, a standardized extraction procedure was followed but different extractants were utilized. All extraction treatments were performed on the same day. DOC, DON and DIN were extracted on 8 replicate samples for each tillage and cropping treatment for each of the 5 extractants used (cold water, hot water,10 mM $CaCl_2$, 2 M KCl and 0.5 M $K_2SO_4$). For each extractant, 5 g of field-moist soil were weighed into acid washed 50 mL polypropylene centrifuge tubes and 25 mL of extracting solution was added (1:5 w/v ratio). All soil suspensions were shaken for 15 min at 200 rpm at 20 °C, except for hot water extractions where the temperature was 80 °C. Soil suspensions were then centrifuged for 10 min at 7900 ×*g-force* at a temperature of 20 °C. Supernatants were filtered through cellulose filter paper (Whatman 42, pore aperture 2.5 μm). One filter paper was used for each supernatant sample and each filter was pre-rinsed with 200 mL ultra-pure water prior to supernantant filtration. Blanks and controls were prepared by filtering water or the respective extractant solution through Whatman 42 filter paper. Sub-samples of soil for each extractant, cropping and tillage combination were oven dried to assess moisture content, which averaged $6.34 \pm 0.73\%$ ($n = 160$). All extract data is reported on an oven-dried soil basis.

### 2.4. Soil leaching

Repacked micro lysimeters or soil columns of each treatment combination were prepared as follows: Columns were 70 mm long, with 27.4 mm internal diameter, and 15 g of 2-mm sieved soil were packed to a bulk density of 1.27 g $cm^{-1}$. Packing comprised 0.5 cm of quartz sand, followed by 15 g of soil sample. A disk of filter paper (Whatman 42, pore aperture 2.5 μm) was placed at the top and bottom of each column and between the soil and sand layers to prevent plugging (Fig. 1).

Prior to the experimental procedure, soil columns were saturated with hot distilled water, from the bottom to top, with the assumption that it would prevent air entrapment, stagnant sections and guarantee a uniform flow domain. Once the columns were saturated, they were maintained immobile for 24 h, to achieve a steady state condition. The flow was then changed to the top from the bottom of columns, and columns were leached with cold distilled water ($EC < 2$ μS) at a 1.5 mL $min^{-1}$ flow rate. Leaching was continued until the electrical conductivity of each column was stable. The volume of leachate from each column was measured at the end of every leaching event. The number of leaching events (pore volumes of water added) for each column varied according to the time taken to reach stabilized electrical conductivity values. Electrical conductivity is one of the indicator variables for the displacement of solutes. Both electrical conductivity (EC) and pH were monitored in the leachates.

### 2.5. Chemical analysis

For all extracts and leachates, DOC and total dissolved nitrogen (TDN) were measured using high temperature platinum-catalyzed combustion with a Shimadzu TOC-VCSH and Shimadzu total measuring unit TNM-1 (Shimadzu Corp. Houston, TX, USA). Dissolved organic carbon was measured as non-purgeable carbon using USEPA method 415.1 which entails acidifying the sample and sparging for 4 min with C-free air. Extracts, but not leachates were further analyzed for ammonium-N using the phenate hypochlorite method with sodium nitroprusside enhancement (USEPA method 350.1) and nitrate was analyzed using Cd–Cu reduction (USEPA method 353.3). Both colorimetric methods were performed with a Westco Scientific Smartchem Discrete Analyzer (Westco Scientific Instruments Inc. Brookfield, CT, USA). DON was estimated as the product of TDN − ($NH_3$–N + $NO_3$–N). Instrument detection limits for each constituent analyzed were $0.5 \pm 0.2$ mg $L^{-1}$ DOC, $0.05 \pm 0.05$ mg $L^{-1}$ TDN, $0.01 \pm 0.012$ mg $L^{-1}$ $NH_4$–N and $0.01 \pm 0.007$ mg $L^{-1}$ $NO_3$–N. Replicate samples, blanks, NIST traceable and check standards were run every 12th sample to